

BLANK PAGE



Indian Stangard

METHODS FOR DETERMINATION OF DOT RESIDUES IN FOOD COMMODITIES

(First Revision)

UDC 664: 543 [632-95-028 DDT!



, @ Copyright 1983

INDIAN STANDARDS INSTITUTION MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

Indian Standard

METHODS FOR DETERMINATION OF DDT RESIDUES IN FOOD COMMODITIES

(First Revision)

Pesticides Residue Analysis Sectional Committee, AFDC 56

Chairman

DR H. L. BAMI Bungalow No. A, Malkaganj, Delhi

Members

SHRI E. A. ALMEIDA SHRI F. QUADROS (Alternate) SHRI K. D. AMRE

DR J. S. VERMA (Alternate)
DR K. C. GUHA

SHRI P. K. DHINGRA (Alternate) DR S. S. GUPTA DR R. L. KALRA

DR R. P. CHAWLA (Alternate)

DR KALYAN SINGH

DR K. KRISHNAMURTHY

SHRI G. N. BHARDWAJ (Alternate)
DR V. LARSHMINARAYANA

DR R. C. GUPTA (Alternate)
DR J. C. MAJUMDAR
DR V. SRINIVABAN (Alternate)
DR M. S. MITHYANATHA
DR A. L. MOOKERJER

Representing

Hindustan Ciba-Geigy Ltd, Bombay

National Organic Chemical Industries Ltd, Bombay

Central Food Laboratory, Calcutta; and Central Committee for Food Standards, New Delhi

Bayer India Ltd, Thane

Department of Entomology, Punjab Agricultural
University, Ludhiana

C. S. A. University of Agriculture and Technology, Kanpur Department of Food (Ministry of Agriculture),

Directorate of Plant Protection, Quarantine and Storage, Faridabad

Pesticides Association of India, New Delhi

Rallis Agro-Chemical Research Station, Bangalore Cyanamid India Ltd, Bombay

(Centinued on page 2)

Copyright 1983

New Delhi

INDIAN STANDARDS INSTITUTION

This publication is protected under the *Indian Copyright Act* (XIV of 1957) and reproduction in whole or in part by any means except with written permission of the publisher shall be deemed to be an infringement of copyright under the said Act.

(Continued from page 1)

Members	Representing		
DR S. K. MUKERJEE	Indian Agricultural Research Institute (ICAR), New Delhi		
DR S. K. HANDA (Alternate)			
DR NAGABHUSHAN RAO	Regional Research Laboratory (CSIR), Hyderabad		
PUBLIC ANALYST	Public Analyst, Government of Haryana, Chandigarh		
DEPUTY PUBLIC ANALYST (Alle			
DR T, D. SETH	Industrial Toxicology Research Centre (CSIR), Lucknow		
DR R. K. SETHI	Indofil Chemicals Ltd, Thane		
DR K. SIVASANKARAN DR S. Y. PANDEY (Alternate)	Union Carbide India Ltd, New Delhi		
DR S. C. SRIVASTAVA	Indian Veterinary Research Institute (ICAR), Izatnagar		
DR R. C. NAITHANI (Alternate)	· ·		
DR K. VISHFSWARAIAH	Central Food Technological Research Institute (CSIR), Mysore		
DR J. R RANGASWAMY (Alterna	te)		
Dr B. L. WATTAL	National Institute of Communicable Diseases, Delbi		
Suri G. C. Josui (Alternate)			
Shri T. Purnanandam, Director (Agri & Food)	Director General, ISI (Ex-officio Member)		
	Secretary		

SHRI M. L. KUMAR
Senior Deputy Director (Agri & Food), 181

AMENDMENT NO. 1 NOVEMBER 1984

TO

IS:5864-1983 METHODS FOR DETERMINATION OF DDT RESIDUES IN FOOD COMMODITIES

(First Revision)

Corrigenda

(Page 10, clause 4.3.2, line 1) - Delete 'l µg/ml each of'.

(Page 11, clause 4.4, line 3) - Substitute 'l ng' for 'l µg'.

(Page 12, clause 4.7, line 2) - Delete 'in 4.7.1' and put a full stop point (.) after 'given'.

(Page 13, clause 5.7, line 1) - Substitute '4.7' for '4.7.1'.

(Page 14, clause 6.4, line 3) - Substitute 'heat' for 'beat'.

(AFDC 56)

Indian Standard

METHODS FOR DETERMINATION OF DDT RESIDUES IN FOOD COMMODITIES

(First Revision)

0. FOREWORD

- 0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 30 August 1983, after the draft finalized by the Pesticides Residue Analysis Sectional Committee had been approved by the Agricultural and Food Products Division Council.
- **0.2** DDT formulations are popular in agriculture and public health for the control of various insect pests and vectors of diseases. Frequent and increased use of these formulations often result in harmful effect due to toxic nature of their residues. Careful assessment of residues is therefore, an important step in safeguarding human health and in the establishment of sound regulatory policy.
- 0.3 This standard was first published as IS:5864 (Part 1)-1970* covering determination of DDT residues in food grains and their milled products. The present revision includes other food commodities not covered earlier and incorporates more sensitive gas chromatographic method. For qualitative confirmation of residues, thin layer chromatography has also been prescribed.
- **0.4** This standard will enable the health authorities and others to follow uniform test procedure for the estimation of DDT residues in various foods.
- **0.5** In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS: 2-1960†.

^{*}Method for determination of DDT residues: Part ! Foodgrains and their milled products.

[†]Rules for rounding off numerical values (revised).

1. SCOPE

- 1.1 This standard prescribes gas chromatographic and spectrophotometric methods for the determination of DDT [2, 2-bis (p-chlorophenyl)-1, 1, 1-trichloroethane and related isomers] residues in foods.
- 1.1.1 The gas chromatographic method determines residues of p,p'-DDT and related isomers such as p,p'-DDE [2,2-bis (p-chlorophenyl) 1-1-dichloroethylene], p,p'-TDE [2,2-bis (p-chlorophenyl) 1,1-dichloroethane] and o,p'-DDT [2-(p-chlorophenyl) 2-(o-chlorophenyl)-1,1,1-trichloroethane] in the range of 0.01 to 0.02 $\mu g/g$ (0.01 to 0.02 ppm), whereas the limit of detection with spectrophotometric method is 0.1 to 0.3 $\mu g/g$ (0.1 to 0.3 ppm).
- 1.1.2 The procedures based on the use of alternate GLC column, thin layer chromatography and microalkali derivatization are also being prescribed for the confirmation of the nature of DDT residues.

2. SAMPLING

2.1 The respresentative sample for the purpose of DDT residues in foods shall be in accordance with the sampling procedures as prescribed in the relevant Indian Standards, wherever available.

2.2 Preparation of the Laboratory Sample

2.2.1 Cereals, Pulses, Spices, Condiments, Tea, Coffee, Sugar Salt, etc — If needed grind to pass through 750 μm (20 mesh) sieve. Reduce to about 100 g by mixing and quartering.

2.2.2 Vegetables and Fruits

- a) Remove visible soil particles by gentle rubbing. Root vegetables may need scrubbing with a brush and some washing.
- b) Remove all inedible portions of vegetables, such as outer leaves of cabbage, carrot tops, stems of tomatoes, stems and stones of fruits, etc.
- c) If a peel or skin is not normally consumed, it should be removed (for example, peas, oranges) and if edible, then it should be included (for example, carrot, potatoes).
- d) Cut a representative sample into pieces and reduce by mixing and quartering to about 300 g.
- e) Blend the 300-g sample in a waring blender to obtain homogeneous mixture. Mix contents with spatula and reblend to ensure homogeniety.

2.2.3 Oilseeds and Nuts

- a) Remove and discard shells, if present.
- b) Reduce the sample by mixing and quartering to the desired sample size needed for extraction. Chop finely,

2.2.4 Fats as Butter, Ghee, Cream, etc

- a) Melt the sample and filter to separate oil from other solids which may be present.
- b) Store the sample in a refrigerator.
- c) Cut the sample into small cubes.
- d) Place cubes (weighing 25 g) in a beaker and heat in an oven at 50°C for the oil to separate.
- 2.2.5 Vegetable Oils (Hydrogenated and Unhydrogenated) These do not need any sample preparation.
- 2.2.6 Milk and Curd Check the homogeneity of the sample. If it is not homogeneous, shake gently.
- 2.2.7 Cheese Chop finely and reduce the sample by mixing and quartering to the desired sample size.

2.2.8 Meats

- a) Remove the inedible portions like head, scales, bones, etc.
- b) Homogenize the composite sample in a waring blender.
- 2.2.9 Eggs Discard shells and examine combined yolks and whites.
- 2.2.10 Water and Beverages These do not need any processing.
- 2.3 Storage of Samples Store samples as such or if the bulk is too much, after preparation of the laboratory sample. Depending upon the nature, store the samples either in deep-freezer (-15°C) or in refrigerator (4°C) until taken up for analysis. Ensure that the samples do not absorb or lose moisture during storage. Avoid undue long storage periods.

3. EXTRACTION AND CLEAN UP

3.1 Apparatus

3.1.1 Blender — Waring or equivalent with glass jar.

- 3.1.2 Centrifuge Centrifuge bottles of at least, 100-ml capacity and capable of operating at 3 000 rev/min.
 - 3.1.3 Extraction Column For clean up of fatty foods (Fig. 1).

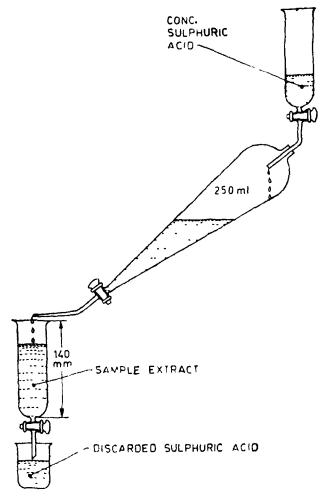
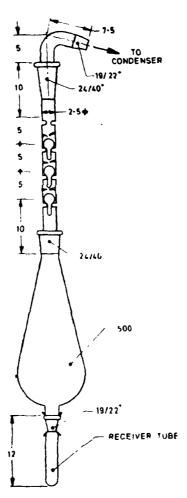


Fig. 1 Extraction Column for Clean up of Sample Extracts

- 3.1.4 Kuderna-Danish Evaporative Concentrator (Fig. 2).
- 3.1.5 Micro-Snyder Column



All dimensions in centimetres.

Fig. 2 Kunderna-Danish Evaporative Concentrator

3.1.6 Rotary Vacuum Evaporator - With 100- and 500-ml concentration flasks.

3.2 Reagents

3.2.1 Sodium Chloride

- 3.2.2 Sodium Sulphate-anhydrous
- 3.2.3 Solvents (acetone, methyl cyanide and n-hexanc) All redistilled in all glass apparatus.
 - **3.2.4** Sulphuric Acid (r.d.1.84)

Note - Check the suitability of the reagents for residue analysis by running reagent blanks.

3.3 Extraction and Partitioning

- a) As the extraction and partitioning schemes are based on moisture, fat and sugar content of the foods, it is suggested that 'The Nutritive Value of Indian Foods and the Planning of Satisfactory Diet' by W.R. Aykroyd, published by the Indian Council of Medical Research, New Delhi or other suitable food composition tables may be consulted to find moisture, fat and sugar content of various food commodities.
- b) If some sample extracts are difficult to filter (wheat flour, rice, bananas) or there is an emulsion formation during partitioning, obtain a clear solution by centrifuging at 3000 rev/min for 10 minutes or more.
- 3.3.1 Non-Fatty Foods Less than 2 percent fat content.
- 3.3.1.1 Commodities with more than 75 percent moisture and less than 5 percent sugar content For example most of the vegetables and some of the fruits. Extract 50 g sample twice with 100 ml and 50 ml methyl cyanide by blending for 3 minutes each. Filter the extracts through Buchner funnel using Whatman No. 1 or equivalent filter paper under vacuum. Transfer the filterate into 1-little separatory funnel, dilute with 600 ml of 5 percent aqueous sodium chloride and partition twice with 100 ml portions of n-hexane. Combine n-hexane layer, dry over anhydrous sodium sulphate and concentrate on a rotary vacuum evaporator or Kuderna-Danish evaporator to about 10 ml.
- 3.3.1.2 Commodities with more than 75 percent moisture and sugar greater than 5 percent For example, grapes and banana, etc. Use methyl cyanide: water $(2:1 \ v/v)$ as extraction solvent and proceed as in 3.3.1.1.
- 3.3.1.3 Commodities with less than 75 percent moisture For example cereals, pulses, condiments, tea leaves, coffee and salt. Extract 50 g sample with 100 ml (or enough to thoroughly wet the sample) portions of methyl cyanide: water $(2:1 \ v/v)$ mixture by blending for 3 minutes each and proceed further as in 3.3.1.1.

- 3.3.1.4 Water and beverages For example, drinking water, soft drinks, tea and coffee infusions. Partition twice 1 litre sample with 100 ml portions of n-hexane, combine the n-hexane layers, dry over anhydrous sodium sulphate and concentrate on a rotary vacuum or Kuderna-Danish evaporator to about 10 ml.
 - 3.3.2 Fatty Foods More than 2 percent fat content.
- 3.3.2.1 Oils and Fals Take 15 g sample, divide it into three 5-g sub-samples and dissolve in 100, 15 and 15 ml portions of n-hexane.
- 3.3.2.2 Milk Take 100 ml milk in the blender, add 100 ml each of acetone and n-hexane and blend for 2 minutes. Transfer homogenate into centrifuge bottles and centrifuge at 2 000 rpm for 10 minutes. Take upper organic phase in a 500-ml separatory funnel and re-extract the lower layer twice with 100 ml portions of n-hexane and collect as before. Wash the combined n-hexane phase twice with 200 ml portions of distilled water. Dry the combined upper n-hexane phase over anhydrous sodium sulphate and concentrate to about 100 ml on rotary vacuum evaporator or Kuderna-Danish evaporative concentrator (see Fig. 2).
- 3.3.2.3 Meat, eggs, nuts, oilseeds, curd, cheese, etc Take suitable sample size (10 to 50 g) containing less than 5 g fat and proceed as in 3.3.2.2.

3.4 Clean-up

3.4.1 For Non-Falty Foods — Take the concentrated n-hexane extract (see 3.3.1) in a 25-ml separatory funnel and add concentrated sulphuric acid dropwise with a pipette till the upper n-hexane layer becomes clear. Discard the lower phase of spent sulphuric acid and wash the upper layer with three 10-ml portions of distilled water. Concentrate the n-hexane phase to the desired volume using either Micro-Snyder column or gentle steam of nitrogen. In general, 50 g samples of cereals, pulses, vegetables, fruits and 1 litre water and beverages will require about 15 ml sulphuric acid.

3.4.2 Fatty Foods

3.4.2.1 Oils and fats — Take the 5 g sub-sample dissolved in 100-ml n-hexane (see 3.3.2.1) in extraction column (see Fig. 1) and add 100-ml sulphuric acid dropwise. Discard lower phase of used sulphuric acid occasionally. Then add to the column the second sub-sample in 15-ml n-hexane, followed again by the dropwise addition of 100 ml of concentrated sulphuric acid. Repeat this step with the third 5 g sample. Discard the lower phase completely, and wash the upper phase with

distilled water till neutral to litmus. Dry over anhydrous sodium sulphate and concentrate to suitable volume.

3.4.2.2 Fatty foods other than oils and fats — Take the n-hexane extract (see 3.3.2.2 and 3.3.2.3) in extraction column (see Fig. 1) and add 100 ml sulphuric acid dropwise. Discard the lower phase completely. Wash, dry and concentrate the upper layer as in 3.4.2.1.

4. GAS-CHROMATOGRAPHIC METHOD

4.1 Principle — The extracted DDT residue are measured using a gas-chromatograph equipped with electron capture detector. The amount of the residues is measured by comparing the sample response with the response of the standards.

4.2 Apparatus

- **4.2.1** Gas-Chromatograph Appropriate gas-chromatograph equipped with electron-capture detector (³H or ⁶³Ni). The suggested operating conditions are:
 - a) Column A column made of borosilicate glass, 1.8 m long, 4 mm I.D. and packed with any of the appropriate materials given below or their equivalents:
 - 1) 1.5 percent OV-17/1.95 percent OV-210 liquid phases premixed and coated on silanized Gas Chrom Q 80/100 mesh.
 - 2) 4 percent SE-30/6 percent OV-210 liquid phases premixed and coated on silanized Gas Chrom Q 80/100 mesh.

b) Injection port temperature : 210°C

c) Column over temperature : 190°C

d) Detector temperature : 220°C

e) Carrier gas (flow rate) : Ultra-high pure nitrogen,

70 ml/min

4.3 Reagents

- **4.3.1** Working standard mixture containing p,p'-DDE, p,p'-TDE o,p'-DDT and p,p'-DDT at the concentration of 0.2, 0.4, 0.4 and 0.4 μ g/ml respectively in n-hexane.
- **4.3.2** Solution containing p,p'-DDE 1 μ g/ml each of at the concentration of 0.2 μ g/ml in n-hexane.

- **4.3.3** Standard mixture containing $|\mu g|$ ml each of p,p'-DDE, p,p'-TDE o,p'-DDT and p,p'-DDT in acetone.
- 4.4 Procedure The column is conditioned and evaluated following the standard procedure, before actual use for analysis. The operating parameters are set to obtain about half-scale deflection with 1 µg p,p'-DDE (see 4.3.2). Inject standard mixture with the component pesticides (see 4.3.1) at three concentration levels with a microlitre syringe. Inject 3-8-µl of the clean-up sample extract so that the injection volume contains about 400 mg sample equivalents of water and beverages or 20 mg of all other samples. If necessary, dilute the extract suitably so that the peak heights of sample and standards do not vary more than 25 percent. Identify the residues by comparing the retention times of the sample peaks with those of the pesticides standards.

4.5 Calculation

DDT residues
$$\mu g/(ppm) = \frac{Hs}{Hstd} \times \frac{M}{M_1} \times \frac{V}{V_1} \times f$$

where

Hs=peak height of the sample;

IIstd=peak height of the standard;

 $M = \mu g$ of standard injected;

 $M_1 = \text{mass}$, in g, of the sample;

V=volume of final extract in ml;

 $V_1 = \mu l$ of the sample injected; and

$$f$$
=recovery factor = $\frac{100}{\text{percent mean recovery}}$

4.6 Expression of Result — Express DDT residues as the sum of p,p'-DDT, o,p'-DDT, p,p'-DDE and p,p'-TDE in the sample as $\mu g/g$ (ppm).

Note — Quantify only the peaks which give greater than 10 percent full-scale deflection (FSD) when the sample equivalent to 400 mg in the case of water and beverages and 20 mg in the case of others, are injected in GLC. Indicate the compounds which produce peaks less than 10 percent FSD as tract (T). If at the retention time of compound, signal is not distinguishable from background, indicate it as not detectable (ND).

4.7 Assuring the Reliability of the Analysis — Pesticides residue analysis involves the determination of the minute quantities of the various compounds in several substrates. Such trace analysis has certain inherent

difficulties and the reliability of the analytical method should be assured by following the procedure given in 4.7.1. Take four samples of the size as recommended under 3.3 and fortify three of these at the level of 2.5 ppm in case of water and beverages, and 0.05 ppm in the case of other commodities by adding appropriate volume of standard mixture (see 4.3.3). Process all the 4 samples following the suitable procedures (see 3 and 4). The recovery of various residues over the background values obtained from unfortified sample should be above 80 percent of the added compounds. The difference between the recoveries from the fortified replicates should be \pm 20 percent.

Note — If the background values of the residues in the sample are expected to be high, increase the level of fortification to about double of the expected background levels.

5. SPECTROPHOTOMETRIC METHOD

5.1 Principle — The cleaned up sample extract is nitrated with nitric acid-sulphuric acid mixture to obtain tetranitro derivative which is then extracted in ether-petroleum ether mixture, dried, taken in benzene and treated with sodium methoxide to get blue colour whose absorbence is measured at 600 nm. The amount of DDT in sample is calculated from the standard curve prepared from pure $\rho_1 p'$ -DDT.

5.2 Apparatus

5.2.1 Spectrophotometer

5.3 Reagents

- 5.3.1 Benzene -- Thiophene Free
- 5.3.2 Ether-Petroleum Ether Mixture Prepare by mixing one part of peroxide and aldehyde free ether with four parts of redistilled petroleum ether.
- 5.3.3 Nitrating Mixture Prepare fresh mixture everytime by mixing equal volumes of fuming nitric acid (r.d. 1.5) and sulphuric acid (r.d. 1.84).
 - 5.3.4 Oleic Acid
 - 5.3.5 Reference Standards
 - 5.3.5.1 10 μ g/ml ρ , ρ' -DDT in benzene.
 - 5.3.5.2 $10\mu g/ml p p'-DDT$ in acetone.

- 5.3.6 Sodium Hydroxide 10 percent aqueous solution.
- 5.3.7 Sodium Methoxide 10 percent, prepare by refluxing 20 gm freshly cut sodium with 500 ml anhydrous methanol; cool, centrifuge and dilute the supernatant with anhydrous methanol. Adjust batches of sodium methoxide to exact concentration by titration with standard acid. Store in dispensing systems excluding carbon dioxide and moisture. Prepare new standard curve for every batch of sodium methoxide.
- 5.4 Preparation of Standard Curve (0 50 µg Range) Take 0, 10, 20, 30, 40, 50 µg p,p'-DDT in benzene (see 5.3.5.1) in 50-ml Erlenmeyer flask, add 10 mg oleic acid in benzene to each flask and evaporate to dryness by a gentle stream of dry air. Chill the flask in an ice-bath and to each add 5 ml chilled nitrating mixture. Swirl the flasks to wet all the portions of the residues and heat on a steam bath for one hour.
- 5.4.1 Cool and transfer the contents of an Erlenmeyer flask to 125-ml separatory funnel using three 20-ml portions of ice cold distilled water. Rinse the flask with two 20-ml portions of ether petroleum ether mixture, add these rinsings to the separatory funnel and re-extract it with 20 ml of ether-petroleum ether mixture. Combine the ether-petroleum ether layers and wash first with two 10-ml portions of aqueous 10 percent sodium hydroxide and then with two 20-ml portions of aqueous saturated sodium chloride. Transfer it to a 250-ml Erlenmyer flask through 1 cm thick layer of anhydrous sodium sulphate previously wetted with etherpetroleum ether mixture. Rinse the separatory funnel with two 10-ml portions of ether-petroleum ether mixture and transfer these rinsings also to the flask. Evaporate the contents in the Erlenmeyer flask to dryness on a steam bath. Cool the flask, dissolve the residue in 3 ml benzene and add 6 ml sodium methoxide. Mix thoroughly and after 15 minutes, measure absorbance at 600 nm against benzene and sodium methoxide (1:2 v/v) solution. Plot absorbance against μg DDT to obtain standard curve.
- 5.5 Quantitation of DDT in the Sample Add 10 mg oleic acid in benzene to the cleaned-up sample extract and evaporate the contents to dryness on a steam bath. Proceed further as for preparation for standard curve as described in 5.4. If absorbance is greater than 0.8, dilute suitably with benzene to get a value in the range of 0.2 to 0.8. Calculate p, p'-DDT in the sample using standard curve. Also measure absorbance at 510 nm and calculate p, p'-DDE using two colour component system.
- 5.6 Expression of Results Express the residues in the samples as $\mu g/g$ (ppm).
- 5.7 Assuring the Reliability of the Analysis Proceed as in 4.7.1 except that fortify the samples at the level of 0.025 ppm in the case

of water and beverages and 0.5 ppm in the case of other commodities by adding appropriate volume of standard solution of p,p'-DDT in acetone (see 5.3.5.2) and process the samples following the suitable procedure outlined in 3 and 5.

6. CONFIRMATION OF THE RESIDUES

- 6.1 The identity of pesticide residues detected and tentatively identified by gas-liquid chromatographic (GLC) method needs confirmation. Depending on the nature and level of the residues, select one or more of the following confirmatory procedures.
- **6.2 Alternate GLC Column** The elution pattern of organochlorine insecticide residues on a column packed with 3 percent DEGS on Gas Chrom Q is quite different from other generally used columns. This column may be used for quick confirmation of the low levels of the insecticide residues.
- 6.3 Thin-Layer Chromatography (TLC) Prepare 0.25 mm thick alumina G plates impregnated with about 40 mg silver nitrate per plate. Air dry a plate, activate it at 110° C for 45 minutes and cool it. Spot insecticide standard mixture (about 0.02 ml of $10 \,\mu\text{g/ml}$ solution of each of p,p'-DDE, p,p'-TDE, p,p'-DDT and o,p'-DDT) and suitable aliquots of cleaned-up extracts. Develop the plate up to 12 to 15 cm in a saturated chamber, using suitable developing solvent, such as heptane, n-hexane or mixture of n-hexane and benzene (4:1 v/v). Air dry and expose the plate to ultra-violent light for about 30 minutes, when the insecticides residues will appear as black spots. Identify the residues by comparing the Rf values with those of the standards co-chromatographed.
- **6.4 Micro-alkali Derivatisation** Take 5 ml aliquot of cleaned-up n-hexane extract having residue concentrations suitable for GLC analysis in a 15-ml graduated tube. Add 2 ml ethanolic KOH and beat in a water bath at 50°C for 2 hours. Add more n-hexane when the solution in the tube nears drying. Remove the tube from the water bath, cool, and add 5 ml ethanol: water $(1:1 \ v/v)$ and 2 ml hexane, stopper and shake gently. Allow the layers to separate and inject suitable aliquots of n-hexane phase into GLC. Conform disappearance or shifts in the peak pattern in alkali derivatized sample as compared to underivatized sample. By this treatment, p,p'-DDE, o,p'-DDT and p,p'-TDE will change to p,p'-DDE, o,p'-DDE and DDMU, respectively, while p,p'-DDE will not be affected.
- 6.5 Interferences Due to Polychlorinated Biphenyls Polychlorinated biphenyls (PCBs) are industrial chemicals which are used in manufacture of synthetic rubber, electrical products, printing inks, paints,

varnishes, resins, etc. These are resistant to oxidation and reduction and thus, like DDT residues, are widely distributed in the environment. Being polychlorinated biphenyls, these have physical and chemical properties of chlorinated bydrocarbon insecucide residues and thus a method of analysis of DDT and BHC will also detect PCBs. In addition to the environmental contamination of the sample, PCBs from rubber and from rubber and plastic caps and lining of the containers and laboratory apparatus can cause interferences. On GLC, PCBs produce multiple peaks some of which may have same retention times as that of BHC and DDT residues. So due care should be taken to avoid the contact of the samples during collection, storage and analysis with rubber or plastics.

6.5.1 If a sample gives multiple peaks on GLC (as will be the case with a sample containing both BHC and DDT residues), the absence or presence of PCBs can be confirmed by micro-alkali treatment (see 6.4).

If after treatment with alkali, the peaks on GLC corresponding to p,p'-TDE, p,p'-DDT and BHC isomers are completely eliminated, it would indicate absence of PCBs. If there is only partial or no decrease in the peaks heights, sample may be containing PCBs. If the suspected PCBs level is fairly high, their presence can be further confirmed by TLC (see 6.3), when the alkali derivatized sample will give at the most 2 spots (one near the solvent and another corresponding to p,p'-DDT), rather than giving several spots corresponding to Rf values of BHC and DDT residues.

INDIAN STANDARDS

ON

PESTICIDES RESIDUE ANALYSIS

	~	
ĸ		

5863 (Part	I)-1970 Method for determination of malathion residues: Part I Cereals and Oilseeds
5864-1983	Methods for determination of DDT residues in food commodities (first revision)
5952-1970	Method for determination of parathion residues in foodgrains and vegetables
6169-1983	Methods for determination of BHC (HCH) residues in food commodities (first revision)
10168-1982	Methods for determination of fenitrothion residues in foods
10169-1982	Methods for determination of carbaryl residues in fruits and vegetables

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUARTITY	Untr	SYMBOL	
Length	metre	m	
Mass	kilogram	kg	
Time	second	•	
Electric current	ampere	A	
Thermodynamic temperature	kelyin	K	
Luminous intensity	candels.	ed	2
Amount of substance	mole	mol	
Supplementary Units		,	
QUANTITY	Unit	SYMBOL	
Plane angle	radian	rad	
Solid angle	steradian	11	
Derived Units			
QUANTITY	Unit	SYMBOL	DEFINITION
Force	newton	N	$1 \text{ N} \Rightarrow 1 \text{ kg.m/s}^{\text{s}}$
Energy	joule	J	1 J == 1 N.m
Power	watt	w	1 W 1 J/s
Flux	weber	₩b	1 Wb == 1 V.s
Flux density	tesla	T	1 T - 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s-1)
Electric conductance	sie mens	S	1.5 = 1.A/V
Electromotive force	volt	v	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

INDIAN STANDARDS INSTITUTION

Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002

Telephones: 26 60 21, 27 01 31 Regional Offices:	Telegrams: Manaksanstha Telephone		
•		•	
Western : Novelty Chambers, Grant Road		32 92 95	
Eastern : 5 Chowringhee Approach	CALCUTTA 700072	27 50 90	
Southern : C. I. T. Campus	MADRAS 600113	41 24 42	
Northern : B69, Phase VII, Industrial Focal Point	S.A.S. NAGAR (MOHALI) 160051	8 78 26	
Branch Offices:			
'Pushpak', Nurmohamed Shaikh Marg, Khanpur	AHMADABAD 380001	2 03 91	
'F' Block, Unity Bldg, Narasimharaja Square	BANGALORE 560002	22 48 05	
Gangotri Complex, Bhadbhada Road, T. T. Nagar	BHOPAL 462003	5 27 16	
22E Kalpana Area	BHUBANESHWAR 751014	5 36 27	
5-8-56C L. N. Gupta Marg	HYDERABAD 500001	22 10 83	
R 14 Yudhister Marg, C Scheme	JA1PUR 302005	6 98 32	
117/418 B Sarvodaya Nagar	KANPUR 208005	4 72 92	
Patliputra Industrial Estate	PATNA 800018	6 28 08	
Hantex Bldg (2nd Floor), Rly Station Road	TRIVANDRUM 695001	32 27	